



Original article

Key composition optimization of meat processed protein source by vacuum freeze-drying technology

Yan Ma^a, Xingzhuang Wu^b, Qi Zhang^a, Vigna Giovanni^c, Xianjun Meng^{d,*}^a Center of Experiment Teaching, Shenyang Normal University, Shenyang 110034, China^b Center of Experiment Teaching, Liaoning Academy of Agricultural Sciences, Shenyang 110161, China^c College of Food, University of Florence, Firenze 50100, Italy^d College of Food, Shenyang Agriculture University, Shenyang 110161, China

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ABSTRACT

Vacuum freeze-drying technology is a high technology content, a wide range of knowledge of technology in the field of drying technology is involved, it is also a method of the most complex drying equipment, the largest energy consumption, the highest cost of drying method, but due to the particularity of its dry goods: the freeze-drying food has the advantages of complex water performance is good, cooler and luster of freezing and drying food to maintain good products, less nutrient loss, light weight, easy to carry transportation, easy to long-term preservation, and on the quality is far superior to the obvious advantages of other dried food, making it become the forefront of drying technology research and development. The freeze-drying process of Chinese style ham and western Germany fruit tree tenderloin is studied in this paper, their eutectic point, melting point and collapse temperature, freeze-drying curve and its heat and mass transfer characteristics are got, then the precool temperature and the highest limiting temperature of sublimation interface are determined. The effect of system pressure on freeze-dried rate in freeze-drying process is discussed, and the method of regulating pressure circularly is determined.

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1. Introduction

Vacuum freeze-dry technology has been applied in the food industry since 1960s. It is beloved all over the world and is considered by many as the best processing method for producing high quality dehydrated food because of its unique advantages which include good re-hydration performance, good color and luster, less nutrient loss, light weight, convenience in transportation, long-term preservation, and far more superior qualities compared with other dried foods (Zhu and Lv, 2011; Feng et al., 2012). According to relevant statistics, the demand for frozen food in the international market is increasing year by year. There was only 200

thousand tons of frozen food produced in the world in 1970s, but the number went up to tens of millions of tons in 1990s (Milford, 2007; Song and Guo, 2013). Freeze-drying food is becoming a popular food in the international market. Therefore, the production of vacuum freeze-drying food has become a hot topic in food processing.

The products produced with the vacuum freeze-drying technology are stored and transported at room temperature, thus no cold chains are needed (Arshadullah et al., 2017). Compared with food products produced with the heated-air drying, vacuum drying, and spray drying methods, those produced with the vacuum freeze-drying method has brighter color and higher nutritional value, and is easier to digest, preserving the nutrients and color, flavor and taste of the meat to the greatest extent (Huan et al., 2005; Kotwaliwale et al., 2007). Therefore, it is especially suitable for drying non-staple food which is extremely sensitive to heat and easy to be oxidized. Freeze drying does not damage the protein in meat and causes no loss of fat-soluble vitamin such as Vitamin A and Vitamin D (Juan and Yuanzhi, 2012; Chen, 2008). By-products and meat processed in this way can totally replace fresh food in terms of nutrition supply.

* Corresponding author.

E-mail address: MENGXJSY@126.COM (X. Meng).

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Therefore, if we apply the vacuum freeze-drying technology in the meat processing industry, it will surely promote this industry's development and drive the development of related industries such as aquaculture and poultry raising, bringing about substantial social value increases (Daya and Pant, 2017). Currently, some domestic enterprises have already adopted this technology (Liu et al., 2014; Gao et al., 2017). After many years of research and development, the Shierde Meat Product Co., Ltd in Tangshan, China has become the first to apply the freeze-drying technology to ham processing, producing high-grade meat products with rich nutrition and a good quality (Tang, 2013).

Development of the technology, mainly adopted vacuum freeze drying technology. Combined with vacuum packing or nitrogen inflator, products have met with potato chips taste and the quality and structure, and the salt content was lower than those of other dry meat, long shelf life. Food freeze drying is one of the most advanced food preservation technology, vacuum freeze drying technology is currently the most advanced drying technology, due to the most complex, the largest energy consumption, drying equipment cost is higher, currently few manufacturers use this kind of drying technology in domestic (Kumruzzaman and Sarker, 2017). At present, for dry meat, a lot of industry use hot drying method, getting the product, color, aroma, taste have great changes, nutrient loss is bigger, after water is poor, not easy to chew. This project adopts the vacuum freeze drying technology. It has many unique advantages, mainly as follows:

- (1) Freeze drying can best preserve meat original color, aroma, taste and nutrients. Freeze drying was conducted under the conditions of low temperature and high oxygen, thus microbes and enzymes have no effect on it, food is also not affected by oxidation, food color, aroma, taste and nutrition loss minimum, so especially suitable for drying food of extremely thermal and oxidation easily. Freeze-dried is not damaging protein in meat, fat soluble vitamins VA, VD, etc have no loss. Nutrition of freeze-dried food that save for 1–2 years can completely take the place of fresh food.
- (2) The freeze drying can best to keep its original form. Food need freezing before dehydrating, forming a stable solid skeleton. Lyophilization was conducted in the frozen state, in the process of freeze-drying, the physical form, chemical and biological properties, solid skeleton basic remain unchanged after dehydration, and can form porous sponge structure, swear, having the ideal instant and fast after water. Freeze-dried meat's after water time is short and water after water also can back into its original shape, after no shrinkage.
- (3) The freeze-dried meat dehydrated thoroughly, had a long shelf life. Freeze-dried food's residual moisture is from 2% to 2%, and the internal residual moisture is well-distributed, water is a necessary condition for the growth of bacteria breeding, bacteria can grow by anhydrous, so in the packaging or vacuum nitrogen-filled packaging and avoid light preservation conditions, so that the fat is not easy to oxidant, prolong storage life and it can be preserved in normal temperature environment for years without deterioration. Freeze-dried product's quality is light, so the sales can be stored at room temperature, very good for marketing. Compared with the frozen food, dried food avoids consumption of high cold chain during the transportation in the process of storage and sales.
- (4) Because the material was frozen in advance, the original solute of inorganic salt dissolved in water was fixed, therefore, solute migration phenomenon is not going to happen when dehydrating, causing surface to harden.

2. Materials and methods

2.1. Materials

Ham and tenderloin were purchased from Jiangsu Yurun Meat Industry Group Co., Ltd. (Nitrates was replaced by monascus pigment as the meat color agent).

2.2. Main instruments and equipment

Scientz-12N vacuum freeze-dryer: Ningbo Xinzhi Instrument Co., Ltd.; Biochrom30 amino acid analyzer: Ningbo Aopu Instrument Co., Ltd.; GT3 texture instrument: United States BIO-RAD Industrial Co., Ltd.; XMT digital regulator: Detection Instrument Factory of Yuyao City, Zhejiang Province; GDM450 digital multimeter: Xi'an Feiteng Instrument Co., Ltd.; Thermocouple thermometer: Beijing Bo Medical Technology Company; FA electronic balance: Shanghai Jing Branch Industrial Co., Ltd.; FDCS196 low temperature freeze-drying microscope: Beijing Aidi Technology Development Co., Ltd.

2.3. Methods

2.3.1. Technical process and experiment

Raw material (ham or tenderloin) → Pre-treatment (Selection Segmentation) → Pre-freezing → Quick Freezing Vacuum Freeze-drying Vacuum or nitrogen packing

2.3.2. Determination of the pre-freezing temperature, co-melting point and eutectic point

The co-melting point and the eutectic point were determined by the resistance method (Li, 2012; Yang et al., 2017), and the test devices are shown in Fig.1. Two mutual insulated copper conductors of appropriate thickness were inserted into the middle of the ham (tenderloin) in parallel, and a copper resistance thermometer was inserted nearby, then the devices were put into the freeze-drying room for pre-freezing. A digital multimeter was used to continuously measure the resistance value of the sample in the pre-freezing process (Ong et al., 2017). The temperature of the samples was also recorded. As the temperature decreased, the resistance value increased. When the temperature reached a certain point, the resistance value suddenly increased to infinity, and this temperature was the eutectic point of the sample. Now, all the material was frozen into a solid, the resistance value reached to maximum because the charged ions were no longer moving. And then the temperature began to increase. The resistance value suddenly became smaller when the temperature reached a certain temperature, which was the co-melting point of the sample.

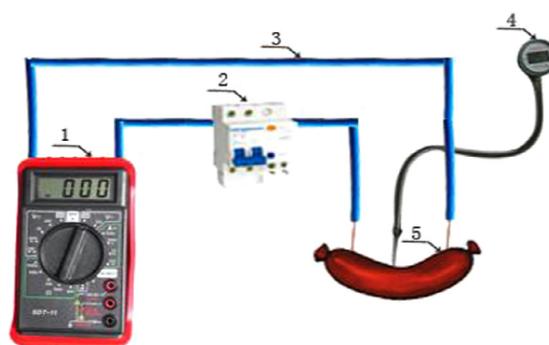


Fig. 1. Schematic of the co-melting point determinator. 1- digital multimeter; 2- switch; 3- wire; 4- resistance thermometer; 5- material.

2.3.3. Determination of the maximum temperature (collapse temperature) on the sublimation interface

The critical temperature was determined using a freeze-drying microscope (FDCS196, Linkam) equipped with a liquid nitrogen cooling system and a vacuum pump. The heating and cooling rate could be kept at 0.01–130 °C /min, and the minimum pressure could reach 0.5 Pa. When the temperature of the lyophilized sample decreased from 25 °C to a temperature below the eutectic point at a rate of 5 °C /min, the sample was evacuated, and the electric valve was adjusted to keep the pressure at 1 Pa for 5 min. Then, the temperature was raised at a rate of 0.5 °C /min. The images were collected using CCD (Retiga2000R, Q Imaging). Through direct observation with the freeze-drying microscope, the collapse temperature of the ham was -31.9 °C and that of the tenderloin was -30.8 °C.

2.3.4. The impact of pressure on the freeze-drying process

The curve of the water content of the material under different pressure (with ham as an example) was measured with the cyclic pressure method. In the first 6 h of the drying process, the system pressure was kept at 10 Pa. From the 6th to the 9th hours of the drying process, the pressure was cyclically alternating, i.e., the pressure was switching between low (10 Pa, 10 min) and high (160 Pa, 10 min) degrees alternately. The system pressure was 10 Pa between 9–10 h. The curve change of the water content of the material was measured at system pressures of 20 Pa and 10 Pa, respectively. The GB5009.3-2010 direct drying method was used to determine water content.

2.3.5. Freeze-drying curve drawing

A whole ham and a whole tenderloin, both with a diameter of 20 mm, were used as the materials. The pre-freezing temperature was -52 °C. The temperatures of the radiation plate, the material center and the material surface at different freeze-drying time were measured, respectively and were used to draw relation curves between the above three temperatures and time.

2.3.6. Single factor experiment

The desorptive drying temperatures (20, 30, 40, 50, 60 °C), the desorptive drying time (2, 3, 4, 5, 6 h), the material thickness (1, 2, 3, 4, 5 mm), and the material size (1/4, 1/2, full piece) were selected as impact factors. Hams (tenderloins) with the same water content were freeze-dried to have a water content of 4%, and the freeze-drying rate (1 mm each freeze time) was used as the measuring indicator.

2.3.7. Orthogonal experiment design

According to the results of the single factor experiment, the appropriate levels of sensory physical and chemical quality of the freeze-dried leisure meat products were selected to carry out the orthogonal experiment, and the range analysis of physical and chemical quality was conducted to get the best process conditions. The rehydration of the lyophilized samples was determined with the gravimetric method. The texture indexes of the freeze-dried and rehydration samples, including fracture, hardness deformation, elastic length, hardness, elasticity and rehydration rate, were measured with a texture analyzer.

2.4. Data analysis

Results were expressed as mean \pm standard deviation ($n = 3$). The difference significance was analyzed with the ANOVA test in the SPSS 17.0 software. When $P < 0.05$, the difference was significant; when $P > 0.05$, the difference was not significant.

3. Results and discussion

3.1. Determination of the pre-freezing temperature, the eutectic point and the co-melting point

It can be seen from Fig. 2 that the co-melting point of the ham and that of the tenderloin were -38 °C and -37 °C, respectively, and their eutectic points were both -42 °C. Freeze-dried products must be frozen to a certain temperature (pre-freezing temperature) before sublimating, and the temperature should be set 10–20 °C below the eutectic point of the product (Li, 2012; D'Agostino et al., 2016). To save energy, the highest pre-freezing temperature that met the requirements was selected, which was -52 °C.

3.2. Determination of the maximum limiting temperature (collapse temperature) of sublimation interface

The collapse of dried product affected the steam pass of the under layer frozen product, so the sublimation rate slowed down and the heat absorption of frozen product decreased, and the heat from the lamina was excessive, which resulted in an increase in the frozen product temperature, and melting foam of the product occurred (Pikal, 2010). As shown in Figs. 3 and 4, the structure of the drying and the frozen areas of the ham samples remained intact at -35.5 °C, and no collapse was found; when the temperature reached -33.6 °C, collapse occurred, and small holes were found in the drying area close to the sublimation interface, which were not connected to each other. When the temperature reached -31.9 °C, all the drying areas close to the sublimation interface collapsed (Sharma et al., 2017). Small holes with no collapse of drying and frozen areas of tenderloin samples were found at -34.7 °C. When the temperature reached -30.8 °C, all the drying area close to the sublimation interface collapsed. Therefore, the collapse temperature of ham was -31.9 °C and the collapse temperature of tenderloin was -30.8 °C.

3.3 Cycle pressure Determination

The freeze-drying process is a process accompanied with simultaneous heat transfer and mass transfer. The lower the system pressure, the faster the mass transfer through the drying layer. However, the decrease of the pressure reduced the effective thermal conductivity coefficient of the drying layer and the heat transmission rate decreased, which indicated that heat and mass transmission were contradictory in the freeze-drying process. In the desorptive phase, because the material did not melt, fast heating was conducted on the material. As the lower pressure did not

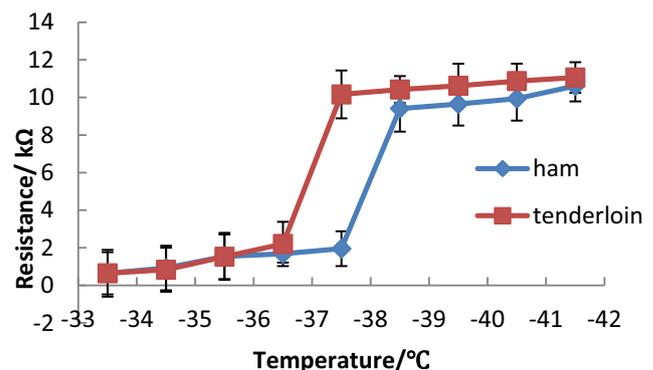


Fig. 2. The impact of temperature on the resistance of sample.

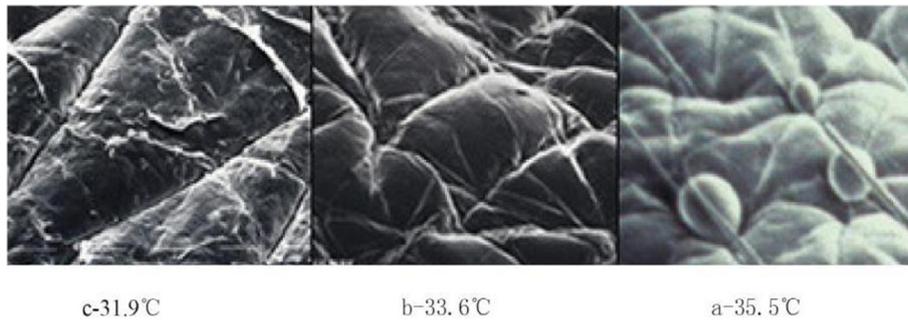


Fig. 3. Microscopy images of the freeze-dried ham (size 100 μm).

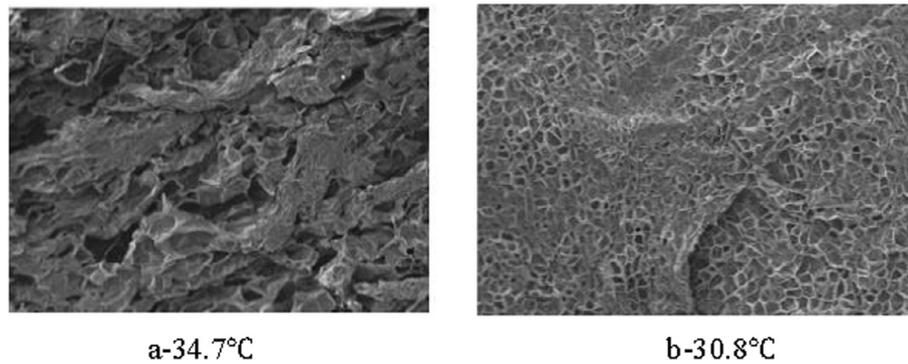


Fig. 4. Microscopy images of the freeze-dried tenderloin (size 100 μm).

meet the requirement, so the circular variable pressure was used in the experiment.

It can be seen in Fig. 5 that all the water content rates decreased rapidly as the freeze-drying time increased under the three pressure conditions. The water content of the sample under lower system pressure was higher than that of higher system pressure, which was beneficial to material drying. Moreover, the circular variable pressure was more conducive to shortening the freeze-drying time and reducing energy consumption.

3.4. Sample freeze-drying curve

The vacuum freeze-drying stage is usually divided into two stages, namely the sublimation drying stage and the desorptive

drying stage. Sublimation drying removes free water from the material, accounting for about 80–90% of the total water content, which is the main body of the freeze-drying process. Desorptive drying ensures the removal of adsorption water strongly bound with solid materials, which accounts for about 10% (Zuo et al., 2010; Li and Wang, 2010; Monteiro, 2008) of the total. It can be seen in Figs. 6 and 7 that 1–6 h was the sublimation drying stage, and the temperature increased slowly in the drying process. 6–11 h was the desorptive drying stage, the temperature increased rapidly, and the final temperature was 60 °C. The temperature of the center of the material was almost unchanged in the early stage, but increased rapidly when the material entered the desorptive drying stage. This was because the temperature of the sublimation interface needed to be carefully controlled in the sublimation stage, close to but, cannot exceed the co-melting point to get the maximum driving force of mass transfer. Given the average temperature difference of the material center and surface was small, about 20 °C, we could infer that the freeze-drying process of the

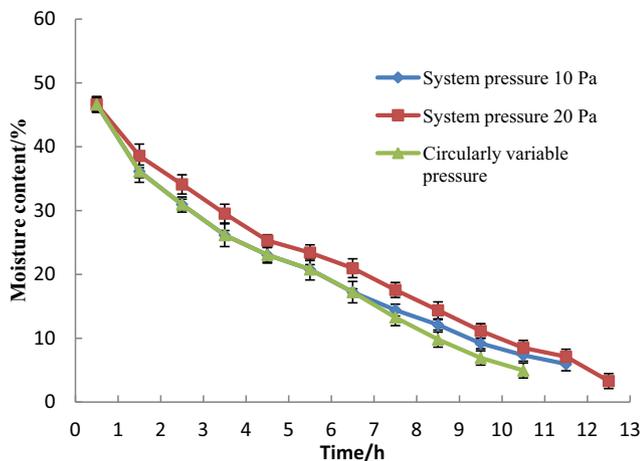


Fig. 5. The impact of pressure on the freeze-drying time of sample.

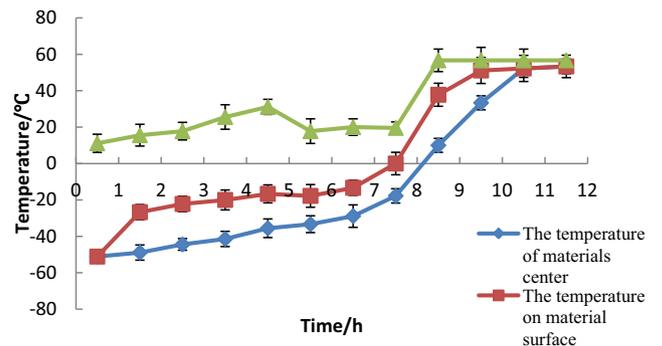


Fig. 6. The freeze-drying curve of ham.

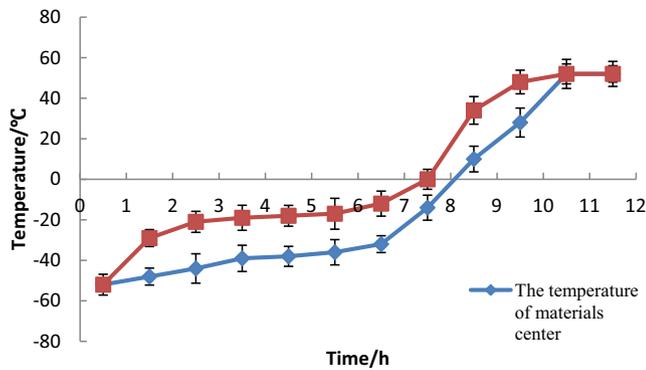


Fig. 7. The freeze-drying curve of tenderloin.

sample was under mass transfer control. Therefore, in the sublimation stage, due to low mass transfer rate, a small difference in the heat transfer temperature could meet the requirements (Daraoui et al., 2010). However, in the desorptive drying phase, as the material would not melt, the temperatures of the material surface and the center could be raised rapidly to the maximum limit temperature to speed up the drying process.

3.5. Single factor experiment results

3.5.1. The desorptive drying temperature

The impact of drying temperature on freeze-drying products was assessed by controlling the drying time for 3 h, material thickness of 3 mm, material size in 1/2, and adjusting the drying temperature at 20, 30, 40, 50, 60 °C. The freeze-drying rate of ham or tenderloin (time for freezing 1 mm each) was selected as the measuring indicator.

Due to the high energy adsorption capacity of adsorptive water, sufficient energy is required to allow them to be desorptive, so the material temperature at this stage should be high enough (Castell-Palou et al., 2011). However, there is a temperature range, and it takes a long time to reach a low temperature; high temperature may result in overheating degeneration. It can be seen in Fig. 8 that the freeze-drying rate of the ham and tenderloin samples increased rapidly with the increase of desorptive drying temperature. When the drying temperature was 50 °C, the freeze-drying rate reached the maximum value; but when the desorptive drying temperature was higher than 50 °C, the freeze-drying rate decreased. Therefore, 50 °C was selected as the best desorptive drying temperature of ham and tenderloin.

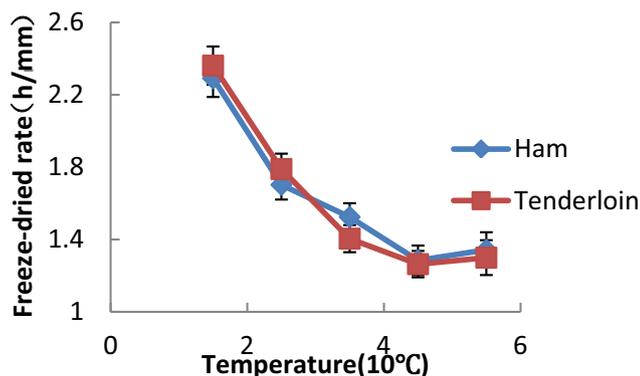


Fig. 8. The impact of temperature on freeze-drying rate.

3.5.2. The desorptive drying time

The impact of the drying time on freeze-drying products was assessed by keeping the drying temperature at 40 °C, the material thickness at 3 mm, the material size at 1/2 of the full piece, and adjusting the drying time to 2, 3, 4, 5, 6 h. The freeze-drying rate of ham or tenderloin (time for freezing 1 mm each) was selected as the measuring indicator.

As short drying time cannot meet the requirement, and long drying time consumes costs too much energy (Luo et al., 2009), a proper drying time needs to be found. As shown in Fig. 9, the freeze-drying rate of the ham and tenderloin samples increased rapidly with the increase of desorptive drying time. When the drying time was 4 h, the freeze-drying rate reached the maximum value; but when the desorptive drying time was longer than 4 h, the freeze-drying rate decreased. Therefore, 4 h was selected as the best desorptive drying time of ham and tenderloin.

3.5.3. Material thickness

The impact of material thickness on freeze-drying products was assessed by keeping the drying temperature at 40 °C, the drying time as 3 h, the material size in 1/2, and adjusting the material thickness to 1, 2, 3, 4, 5 mm. The freeze-drying rate of ham or tenderloin (time for freezing 1 mm each) was selected as the measuring indicator.

The freeze-drying time of the material is usually extended as its thickness increases, but the freeze-drying time of unit thickness is different (Yang and Liu, 2010; Jaruk and John, 2006). Short drying time cannot meet the requirement, and long drying time takes high energy (Luo et al., 2009), so a proper drying time needs to be found. As shown in Table 3-1, the freeze-drying rate of the ham and tenderloin samples increased rapidly with the increase of material thickness. When the material thickness was 3 mm, the freeze-drying rate reached the maximum value; but when the material thickness was thicker than 3 mm, the freeze-drying rate decreased. Therefore, 3 mm was selected as the best material thickness of ham and tenderloin.

3.6. Orthogonal experiment results of freeze-dried meat products

The expected product is freeze-dried meat product with the potato chips texture, so it will be better when the product hardness, flexibility, viscosity, chewiness, energy consumption is lower and the rehydration rate are higher (Wu and Guo, 2010). And through the determination of texture, we found changes in adhesive was small, so we didn't select it as the main factor for consideration. At the same time, secondary circulation was used to

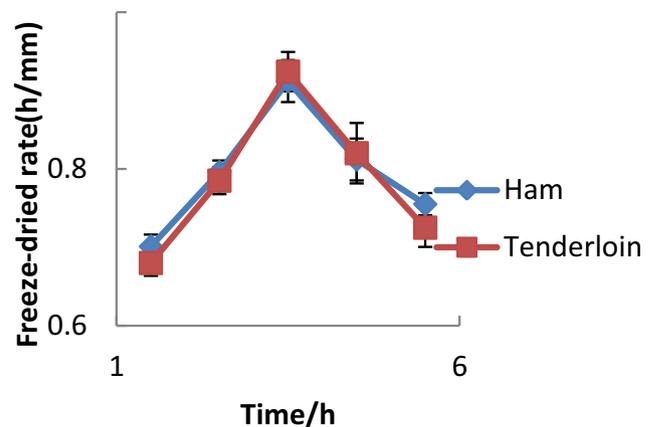


Fig. 9. The impact of time on freeze-drying rate.

Table 3-1

The freeze-drying rate of ham with various thicknesses.

Thickness of sample/mm	1	2	3	4	5
Freeze-drying time of ham/h	1.6	2.5	3.6	5.2	6.6
Freeze-drying rate of ham (h/mm)	1.6	1.25	1.2	1.3	1.32
Freeze-drying time of tenderloin /h	1.5	2.4	3.5	5	6.5
Freeze-drying rate of tenderloin (h/mm)	1.5	1.2	1.17	1.25	1.3

determine the texture and the indicators were comprehensively analyzed.

According to previous results, material thickness, desorptive drying time, desorptive drying temperature and material size were selected as influence factors. The hardness, breakability, shape variable of hardness and elastic length of first circulation, and the hardness and rehydration rate of secondary circulation were used as indicators. L_9 (3^4) orthogonal experiment was conducted. The factors and levels of ham and tenderloin are shown in Tables 3-2 and 3-3, respectively.

As shown in Table 3-4, the primary factor affecting the first cycle hardness and breakability was thickness, and shape and temperature were the secondary factors. The order of the importance of the factors was: $C > D > A > B$, and the primary and secondary relations of the main factors affecting the freeze-dried tenderloin were: material thickness > material size > desorptive drying temperature > desorptive drying time. The best combination of freeze-dried ham was: $A_3B_1C_2D_1$, i.e., the desorptive drying temperature was 55 °C, the desorptive drying time was 3 h, and the material thickness was 2 mm (1/4 slice). The most important factor affecting the hardness deformation was thickness, and shape and time were the secondary factors. The order of the importance of factors was: $C > D > B > A$, the primary and secondary relations of the main factors affecting freeze-dried ham were: material thickness > material size > desorptive drying time > desorptive drying temperature. The best combination of freeze-dried ham was: $A_2B_2C_2D_1$, i.e., the desorptive drying temperature was 50 °C, the desorptive drying time was 4 h, and the material thickness was 2 mm (1/4 slice). The most important factor affecting the elasticity length was desorptive drying time, and shape and thickness were the secondary factors. The order of the importance of factors was: $B > D > C = A$, the primary and secondary relations of the main factors affecting freeze-dried ham were: desorptive drying time > material size > material thickness = desorptive drying temperature. The best combination of freeze-dried ham was: $A_1B_1C_1D_2$, i.e., the desorptive drying temperature was 45 °C, the desorptive drying time was 3 h, and the material thickness was 1 mm (1/2 slice). The most important factor affecting the second cycle hardness was shape; and time and thickness were the secondary factors. The order of the importance of factors was: $D > B > C > A$, and the primary and secondary relations affecting the main factors of freeze-dried ham were: material size > desorptive drying time > material thickness > desorptive drying temperature. The best combination of freeze-dried ham was $A_1B_2C_1D_1$, i.e., the desorptive drying temperature was 45 °C, the desorptive drying time was 4 h, and the material thickness was 1 mm (1/4 slice). The most important factor affecting elasticity was thickness; and shape and time were

the secondary factors. The order of the importance of factors was: $C > D > B > A$, and the primary and secondary relations affecting the main factors of freeze-dried ham were: material thickness > material size > desorptive drying time > desorptive drying temperature. The best combination of freeze-dried ham was $A_3B_2C_1D_1$, i.e., the desorptive drying temperature was 55 °C, the desorptive drying time was 4 h, and the material thickness was 1 mm (1/4 slice). The most important factor affecting rehydration ratio was shape, and thickness and temperature were the secondary factor. The order of the importance of factors was: $D > C > A > B$, and the primary and secondary relations affecting the main factors of freeze-dried ham were: material size > material thickness > desorptive drying temperature > desorptive drying time. The best combination of freeze-dried ham was $A_1B_2C_2D_3$, i.e., desorptive drying temperature was 45 °C, desorptive drying time was 4 h, and material thickness was 2 mm (full piece).

The above indexes were analyzed according to the comprehensive evaluation method. First of all, for Factor A: temperature, it is the most important factor affecting the first cycle hardness, so A_3 was selected as the superior value; its impact on the rehydration ratio was also the most important, so A_1 was selected as the superior value; as their impact on other indicators were secondary, the values A_1 and A_3 values were selected out of A. After a comprehensive evaluation of the impact of A_1 and A_3 on the first cycle hardness, hardness deformation, elasticity length and rehydration ratio, A_3 was selected for having the best level. For Factor B: drying time, it is the most important factor affecting elasticity length, and its impact on the second cycle hardness was also important. As the impact of factor B on the other factors was not significant, B_1 was selected as the best level. For Factor C: material thickness, it is the most important factor affecting the first cycle hardness, hardness deformation and elasticity, so C_2 and C_1 were chosen as the superior values. When the thickness of the material was 3 mm, the first cycle hardness of the freeze-dried ham decreased by 4.49%, the hardness deformation was reduced by 13.06%, and the elasticity increased by 56.4%. Considering the impact of elasticity on the quality of freeze-dried ham, C_1 was selected as the best level i.e., the material thickness was 2 mm. For Factor D: material size, it is the most important factor affecting the second cycle, so D_1 was considered as the best value; as its impact on the rehydration ratio was also important, D_3 was chosen as the superior value; its impact on other indicators were secondary. However, when the material size was 1/4 piece, the second cycle hardness decreased by 74.78%. When the material size was full piece, the rehydration ratio was 6.83% higher than that of the 1/4 material size. When the material size was 1/4, the other indexes were lower than those of full material size. Therefore, D_1 was selected as the best level,

Table 3-2

The orthogonal factor levels of freeze-dried ham.

Levels	Factors			
	Temperature (°C) A	Time (h) B	Thickness (mm) C	Shape (piece) D
1	45	3	1	1/4
2	50	4	2	1/2
3	55	5	3	1

Table 3-3

The orthogonal factor levels of freeze-dried tenderloin.

Levels	Factors			
	Temperature (°C) A	Time (h) B	Thickness (mm) C	Shape (piece) D
1	45	3	2	1/4
2	50	4	3	1/2
3	55	5	4	1

Table 3-4

The orthogonal experiment results and range analysis of freeze-dried ham.

NO.	Factors				Physicochemical indicators					
	A	B	C	D	First cycle hardness, breakability	Hardness deformation	Elasticity length	Second cycle hardness	Elasticity	Rehydration ratio
1	1	1	1	1	212	1.44	0.44	31	0.02	70.02
2	1	2	2	2	458	0.63	0.99	50	0.57	74.32
3	1	3	3	3	283	3.53	1.08	212	3.03	81.54
4	2	1	2	3	304	1.77	0.99	272	1.22	84.47
5	2	2	3	1	669	0.78	2.06	68	1.02	77.24
6	2	3	1	2	139	0.89	0.48	121	0.59	55.75
7	3	1	3	2	867	2.51	0.28	214	1.15	64.32
8	3	2	1	3	239	1.27	1.48	87	0.48	72.73
9	3	3	2	1	432	0.73	0.77	45	0.71	75.18
K1 _F	3333	2681	2404	2390						
K2 _F	3011	2773	2296	3105						
K3 _F	2550	3440	4194	3399						
R _F	195	176.33	409.67	212.67						
K1 _H	5.6	5.72	3.6	2.95						
K2 _H	3.44	2.68	3.13	4.03						
K3 _H	4.51	5.15	6.82	6.57						
R _H	0.72	1.01	1.23	1.21						
K1 _{EL}	2.51	1.71	2.4	3.27						
K2 _{EL}	3.53	4.53	2.75	1.75						
K3 _{EL}	2.53	2.33	3.42	3.55						
R _{EL}	0.34	0.94	0.34	0.51						
K1 _S	293	517	239	144						
K2 _S	461	205	367	385						
K3 _S	346	378	494	571						
R _S	56	104	85	142.33						
K1 _E	3.62	2.39	1.09	1.75						
K2 _E	2.83	2.07	2.5	2.31						
K3 _E	2.34	4.33	5.2	4.73						
R _E	0.43	0.75	1.37	0.99						
K1 _R	225.88	218.81	198.5	222.44						
K2 _R	217.46	223.87	233.97	194.39						
K3 _R	212.23	212.47	223.1	238.74						
R _R	4.55	3.8	11.82	14.78						

i.e., the material size was 1/4. In summary, the best combination of freeze-dried ham was A₃B₁C₁D₁, i.e., ham desorptive drying temperature: 55 °C, desorptive drying time: 3 h, material thickness: 1 mm, and material size: 1/4 piece.

As shown in Table 3-5, the most important factor affecting the first cycle hardness and brity was thickness and the shape and temperature were the secondary factors. The order of the importance of factors was: C > D > A > B. The primary and secondary relations of the main factors affecting freeze-dried tenderloin were: material thickness > material size > analytical drying temperature > analytical drying time. The best combination of freeze-dried tenderloin was A₃B₁C₂D₁, i.e., the desorptive drying temperature was 55 °C, the desorptive drying time was 3 h, and the material thickness was 3 mm (1/4 slice). The most important factor affecting the hardness deformation was temperature, and thickness and shape were secondary factors. The order of the importance of factors was: A > C > D > B. The primary and secondary relations of the main factors affecting freeze-dried tenderloin were: desorptive drying temperature > material thickness > mate-

rial size > desorptive drying time. The best combination of freeze-dried tenderloin was A₁B₁C₂D₁, i.e., the desorptive drying temperature was 45 °C, the desorptive drying time was 3 h, and the material thickness was 3 mm (1/4 slice). The most important factor affecting the elasticity length was temperature and thickness and time were secondary factors. The order of the importance of factors was: A > C > B > D. The primary and secondary relations of the main factors affecting the freeze-dried tenderloin were: desorptive drying temperature > material thickness > desorptive drying time > material size. The best combination of freeze-dried tenderloin was A₁B₂C₁D₂, i.e., the desorptive drying temperature was 45 °C, the desorptive drying time was 4 h, and the material thickness was 2 mm (1/2 slice). The most important factor affecting the second cycle hardness was thickness and shape and temperature were secondary factors. The order of the importance of factors was: C > D > A > B. The primary and secondary relations of the main factors affecting freeze-dried tenderloin were: material thickness > material size > desorptive drying temperature > desorptive drying time. The best combination of freeze-dried ham was A₂B₂C₂D₂, i.e., the

Table 3-5

The orthogonal experiment results and range analysis of freeze-dried tenderloin.

(NO.)	Factors				Physicochemical indicators					
	A	B	C	D	First cycle hardness, breakability	Hardness deformation	Elasticity length	Second cycle hardness	Elasticity	Rehydration ratio
1	1	1	1	1	638	0.33	1.06	130	1.04	71.25
2	1	2	2	2	871	0.88	1.16	43	0.25	81.71
3	1	3	3	3	1824	1.97	1.02	705	1.16	83.62
4	2	1	2	3	831	1.03	1.83	248	0.85	82.5
5	2	2	3	1	1158	1.29	1.63	336	1.33	72.64
6	2	3	1	2	1022	1.24	1.49	28	1.05	67.32
7	3	1	3	2	1212	2.88	2.38	392	1.58	51.14
8	3	2	1	3	744	2.46	1.95	362	1.97	76.67
9	3	3	2	1	594	1.09	2.35	120	1.67	79.12
K1F	3333	2681	2404	2390						
K2F	3011	2773	2296	3105						
K3F	2550	3440	4194	3399						
RF	261	253	632.67	336.33						
K1H	3.18	4.24	4.03	2.71						
K2H	3.56	4.63	3	5						
K3H	6.43	4.3	6.14	5.46						
RH	1.08	0.13	1.05	0.92						
K1EL	3.196	5.27	4.5	5.04						
K2EL	4.95	4.74	5.34	5.03						
K3EL	6.68	4.86	5.03	4.8						
REL	1.61	0.18	0.28	0.08						
K1S	878	770	520	586						
K2S	612	741	411	463						
K3S	874	853	1433	1315						
RS	88.67	37.33	340.67	284						
K1E	2.45	3.47	4.06	4.04						
K2E	3.23	3.55	2.77	2.88						
K3E	5.22	3.88	4.07	3.98						
RE	0.92	0.14	0.43	0.39						
K1R	236.58	204.89	215.24	223.01						
K2R	222.58	231.02	243.33	200.17						
K3R	206.93	230.06	207.4	242.79						
RR	9.88	8.71	11.98	14.21						

desorptive drying temperature was 50 °C, the desorptive drying time was 4 h, and the material thickness was 3 mm (1/2 slice). The most important factor affecting elasticity was temperature and thickness and shape were the secondary factors. The order of the importance of factors was: A > C > D > B. The primary and secondary relations of the main factors affecting the freeze-dried tenderloin were: desorptive drying temperature > material thickness > material size > desorptive drying time. The best combination of freeze-dried tenderloin was A₁B₁C₂D₂, i.e., the desorptive drying temperature was 45 °C, the desorptive drying time was 3 h, and the material thickness was 3 mm (1/2 slice). The most important factor affecting rehydration ratio was shape and thickness and temperature were secondary factors. The order of the importance of factors was: D > C > A > B. The primary and secondary relations of the main factors affecting freeze-dried tenderloin were: material size > material thickness > desorptive drying temperature > desorptive drying time. The best combination of freeze-dried tenderloin was A₁B₂C₂D₃, i.e., the desorptive drying temperature was 45 °C, the desorptive drying time was 4 h, and the material thickness was 3 mm (full piece).

The above indexes were analyzed according to the comprehensive evaluation method. Firstly, for Factor A: temperature, it is the most important factor affecting the hardness deformation, elasticity length and elasticity, therefore, A₁ was selected as the superior value. Meanwhile, the impact of temperature on the other indicators were secondary, so A₁ was selected as the best level. For Factor B: drying time, it is the most important factor affecting elasticity length, and secondary to other factors, so B₂ was selected as the best level. For Factor C: material thickness, it was the most impor-

tant factor affecting the first cycle hardness and the second cycle hardness. Considering the trend of the above two indicators, C₂ was the selected as the superior value. Meanwhile, the impact of factor C on other indicators were secondary, so C₂ was selected as the best level. For Factor D: material size, it is the most important factor affecting the rehydration rate. So D₃ was selected as the superior level. Meanwhile, the impact of factor D on other indicators were secondary, so D₃ was selected as the best level. Based on the above analysis, the best combination of freeze-dried tenderloin was A₁B₂C₂D₃, i.e., the desorptive drying temperature of the ham was 45 °C, the desorptive drying time was 4 h, the material thickness was 3 mm, and whole film (single layer) was tiled.

4. Conclusion

- (1) The co-melting points of the ham and tenderloin were –38 °C and –37 °C, respectively, and their eutectic points were both –42 °C, which were measured with the resistance method. Freeze-dried products must be frozen to a certain temperature (pre-freezing temperature) before sublimating, and the temperature should be set 10–20 °C below the eutectic point of the product. To save energy, –52 °C, the highest temperature that met the requirements, was selected as the pre-freezing temperature.
- (2) The critical temperature (maximum limit temperature of the sublimation interface (collapse temperature)) was determined with a freeze-drying microscope. The collapse temperature of ham was –31.9 °C and that of tenderloin was –30.8 °C.

- (3) In the cyclic pressure alternating experiment, the lower system pressure, the faster the drying process. Moreover, the cycle of alternating pressure was more conducive to shortening the freeze-drying time and reducing energy consumption.
- (4) The freeze-dry curve of the sample showed that the freeze-drying process was under mass transfer control and the sublimation drying time was 5 h. In the sublimation stage, due to the low mass transfer rate, the smaller difference in heat transfer temperature met the requirements. In the desorptive phase, the temperature increased rapidly and made the surface and center temperature reach the maximum limiting temperature as soon as possible, which sped up the drying process.
- (5) In the single factor experiment, with the freeze-drying rate as the measuring indicator, the results showed that the desorptive freeze-drying temperatures was 50 °C, the best desorptive drying time was 4 h, and the optimum freeze-drying thickness was 3 mm. According to the shape characteristics of the raw materials (ham and tenderloin), the product shapes were basically all 20 mm × 20 mm circles, and samples were set at 1/4, 1/2 and 1/1 of the full piece.
- (6) According to the results of the orthogonal experiment and the range analysis, the best combination of freeze-dried ham was $A_3B_1C_1D_1$, i.e., the desorptive drying temperature of ham was 45 °C, the desorptive drying time was 3 h, material thickness was 1 mm, and material size was 1/4piece; the best combination of freeze-dried tenderloin was $A_1B_2C_2D_3$, i.e., the desorptive drying temperature of tenderloin was 45 °C, the desorptive drying time was 4 h, the material thickness was 3 mm, and the material size was full piece (single layer).

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